United States Department of Agriculture
Agricultural Research Service
Southern Utilization Research Branch

NEW DIRECT CONSUMPTION SUGAR

CA-27

Southern Regional Research Laboratory
New Orleans 19, Louisiana
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NEW DIRECT CONSUMPTION SUGAR

Increased Production and Better Quality Obtained by Ion-Exchange Process

Experimental Production and Evaluation for Use in Candy Manufacture

The Ion-Exchange Process

The exchange of bases such as calcium for potassium on natural soil substances was discovered more than a century ago[1]. The earliest attempt to use this property of natural clays was for removal of calcium from beet juice in a process patented by Harm[2] in 1896. This process was not a commercial success. Almost 50 years ago Gans[3] patented the use of natural zeolites for water softening by replacing the calcium and magnesium by sodium, and this has been adopted universally. When resins capable of exchanging acids from solution were synthesized by Adams and Holmes[4] 20 years ago it became possible to extend the use of ion-exchange for water purification and to apply it to many other uses. Hundreds of practical uses for the process have been developed in industry, and domestic units for purifying household water supplies are commonplace in areas where only hard water is available.

Adams and Holmes' discovery led to the rapid development of a host of varied and better synthetic ion-exchange resins. The process is undergoing continual improvement and refinement for an ever increasing variety of industrial applications. Commercial production of more efficient resins for exchanging acids from solutions of salts makes it possible to purify sugarcane juice more efficiently than was possible with the ion-exchange resins available five years ago.

What the Ion-Exchange Process Does

Crystallization of sugar from sirups is limited by the amounts of impurities present, most of which are combinations of mineral and organic acids with bases that form the natural salts present in cane juice. Ion-exchange resins can remove a large proportion of these impurities from the juice before it is concentrated to sirup, making it possible to crystallize more of the sugar. The raw juice must first be treated with lime and heated to clarify it exactly as in the usual production of raw sugar. When the clarified juice is percolated through a column of solid, granular acid exchange resin, the organic and mineral acids are bound by the resin and thus removed from the juice. The juice becomes alkaline, as it contains the free bases. After sufficient juice has percolated through the resin to exhaust its acid-removing capacity, water is passed through to wash out remaining juice, and the activity of the resin is restored by percolating

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1/ Thompson, H. S., J. Roy. Agr. Soc. Engl. 11, 68 (1850); Way, J. T., J. Roy. Agr. Soc. Engl. 11, 313 (1850); Eichorn, E., Pogg. Ann. 105, 126 (1858).
2/ Harm, F., German Pat. 95,447 (1896).
3/ Gans, R., German Pat. 197,111 (1906).
4/ Adams, B. A. and Holmes, E. L., J. Soc. Chem. Ind. 54, 1T (1953).

a solution of caustic soda through the column. The alkaline juice containing free bases is next percolated through a column of base exchange resin that removes the bases, and the juice emerges substantially neutral. This column of resin is then washed free of juice, after which its activity is restored by treatment with a solution of sulfuric acid. The resins can be used over and over again to treat large volumes of juice.

By selecting ion-exchange resins with high capacities for removing the impurities from sugarcane juice, the purity of the sirups can be increased to yield 8 to 10% more crystalline sugar under Louisiana sugar manufacturing conditions.

The Direct Consumption Sugar Process

The simplified flow diagram illustrates the use of ion-exchange resins to obtain high yields of direct consumption sugar from clarified cane juice. The process is an added purification step in the conventional process by which raw sugar is obtained; the product is a practically white sugar suitable for many uses without further purification. It is not as pure as refined sugar produced by redissolving raw sugar and percolating the sirup through granular bone char.

Up to the pre-cooler in the diagram, grinding of cane to extract the juice and its clarification with lime and heating are exactly the same as in the manufacture of raw sugar. The pre-cooler is a vacuum evaporator that reduces the temperature of the clarified juice to about 95° F. by evaporating not more than 10% of the water present. The somewhat concentrated, cooled juice is percolated through a bed of granular acid exchange resin about 4 feet in depth. Rohm and Haas' strong anion exchange resin IRA-410[5] was used in the pilot plant scale experiments, as it is highly basic and can separate acids efficiently from their salts present in the juice. Flow is continued until the change in alkalinity of the outflow juice leaving the bottom of the bed indicates that the resin has combined with as much of the acids in the juice as it can hold efficiently. Water is then percolated through the bed of acid exchange resin until it is free of sugar. Precipitated solids are next washed out by an upward flow of water. This washing is followed by percolating down through the column enough caustic soda solution to remove the acids and restore the activity of the resin. Washing down with pure water to flush out excess caustic soda solution then leaves the regenerated, active resin ready to remove acids from juice in a repetition of the exchange cycle. Several acid-exchange units are used so that the flow of raw juice is continuous, passing through the active beds while others are being regenerated.

The bases are removed from deacidified juice by percolation through beds of a suitable base-exchange resin 4 feet deep. The cycle of operations is similar to that used for the acid-exchange resin beds. As the

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Up to the pre-cooler in the diagram, grinding of cane to extract the juice and its clarification with lime and heating are exactly the same as in the manufacture of raw sugar. The pre-cooler is a vacuum evaporator that reduces the temperature of the clarified juice to about 50° F. by evaporating not more than 10% of the water present. The somewhat concentrated, cooled juice is percolated through a bed of granular acid exchange resin about 4 feet in depth. Rohm and Haas' strong cation exchange resin (IR-100) 3/ was used in the pilot plant scale experiments, as it is highly acidic and can separate acids efficiently from their salts present in the juice. Flow is continued until the change in alkalinity of the outflow juice leaving the bottom of the bed indicates that the resin has combined with as much of the acids in the juice as it can hold efficiently. Water is then percolated through the bed of acid exchange resin until it is free of sugar. Precipitated solids are next washed out by an upward flow of water. This washing is followed by percolating down through the column enough caustic soda solution to remove the anion and restore the activity of the resin. Washing, then with pure water to flush out excess caustic soda solution then leaves the regenerated, active resin ready to remove acids from juice in a repetition of the exchange cycle. Several acid-exchange units are used so that the flow of raw juice is continuous, passing through the active beds while others are being regenerated.

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bases are free, that is, no longer combined with acids in the form of salts, a weakly acidic base-exchange resin can be used. This is also important to avoid inversion of sugar, as will be explained below, and Rohm and Haas resin IRC-50 is used as the base-exchange resin in the pilot plant experiments. In this case the outflowing juice becomes more acid as the capacity of the resin is saturated. After washing the bed free of sugar with water, the base-exchange resin is regenerated with a solution of sulfuric acid. Backwashing of precipitated material and final washing out of excess acid by percolating pure water through the bed prepares the regenerated resin for a repetition of the base-exchange cycle.

A third bed of exchange resins 4 feet in depth is used to obtain additional purification and especially removal of color. Small amounts of acids not removed by the first acid-exchange resins are still present in juice that has passed through the first two exchangers. These are readily removed by an additional percolation through the acid-exchange resin, IRA-410. Because less resin is required for this step it is mixed with another acid-exchange resin that is more efficient for removing colored impurities from the juice. The Chemical Process Company resin A-2 is used in the pilot plant for this purpose. Other combinations of resins have been found effective in this and the other exchange steps of the process in laboratory experiments[6]. Regeneration of this acid-exchange resin bed is carried out in the same manner as in the case of the first acid-exchanger, using caustic soda for regeneration.

The purified juice goes to the evaporators used in the conventional raw sugar process and the sugar can be crystallized by the usual methods in standard vacuum pans. Higher purity of the sirup makes it possible to obtain an additional "strike" or crop of sugar. Making larger grain crystals and washing with steam in the centrifugals yields a product sufficiently pure and white for bagging and marketing for many industrial uses.

Inversion

When solutions of sugar such as cane or beet juice are acidified the ordinary sugar, sucrose, is more or less rapidly broken down into two simpler sugars, glucose and fructose. This reaction is called inversion and the equal mixture of glucose, or corn sugar, and fructose, or fruit sugar, is termed "invert sugar." Invert sugar sirups are used widely in candy manufacture, baking, and other foods. The older types of acid-exchange resins were not effective in removing acids until they had been liberated by strong base-exchange resins. These strong base-exchange resins and the acids liberated by their use caused the inversion of an appreciable amount of the sugar. This proved to be a major technical obstacle to the use of earlier types of ion-exchange resins for purifying sugarcane juice. This

6/ Fort, C. A. and Smith, B. A., Sugar J. 15, 9, pp. 16, 18, 22-25 (1953); Fort, C. A., Smith, B. A., and Martin, L. F., Proc. 3rd Tech. Session on Bone Char, 1953 (May 1954); Fort, C. A. and Smith, B. A., "Ion Exchange. Recovery and Quality of Sugar Produced by Reverse Cycle Purification of Sugarcane Juice." In press. Scheduled for publication in April 1955 issue of Sugar.

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loss of sugar could be minimized by cooling the juice to a sufficiently low temperature before passing it through the ion-exchange system. Low temperature cooling water is available where beet sugar is produced so that ion-exchange has been used commercially in factories where it is economically feasible to cool the beet juice to below 70° F.[7]

Cane sugar is produced under tropical or sub-tropical conditions, where factory cooling water is seldom below 75-80° F. The cost of refrigeration to cool the juice made the process uneconomical with the exchange resins available until 5 years ago. Extensive pilot plant experimentation was carried out on this process with sugarcane juice in Hawaii[8]. Development of more effective acid-exchangers that can be used without exposing the juice to acid conditions makes it possible to purify juice that has been cooled only to 95° F., which is economically feasible in cane sugar factories. Advantages of this method of purifying cane juice were first described in a patent by McBurney[9]. Resins may also be used in the mixed bed process patented by Kunin and McGarvey[10], which is also effective for removing acids and bases from the juice without loss of sugar by inversion. In their method the acid-exchange and base-exchange resins are mixed while the juice is percolated through the bed. After saturation they must be separated by floating the lighter acid-exchange resin to the top, and are then separately regenerated by treatment with caustic soda and acid respectively. An ion-exchange membrane process has been patented[11] that does not involve the use of caustic soda and acid for regeneration. More laboratory research and larger scale experiments will be necessary to determine whether it can be used to purify cane juice industrially.

Small Pilot Plant Experiments

Production of direct consumption sugar from the clarified juice of Louisiana cane has been carried out experimentally during the 1953 and 1954 grinding seasons on a comparatively small scale. The process is still undergoing experimental development, and continuous production in a larger pilot plant will be necessary to establish precisely the cost as well as yield and quality of sugar. Estimates based upon small scale experiments completed thus far are sufficiently favorable to indicate the desirability of further process development work on a larger, continuous pilot plant scale.

The plastic columns used in the experiments are 5-1/2 inches in diameter, and contain about 1/2 cubic foot of exchange resin in each column. Three of

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7/ Vallez, H. A., U. S. Patents 2,388,194 and 2,388,195 (1945); Maudru, J. E., Ind. Eng. Chem. 43, 615 (1951); Sugar 46 (1), p. 28 (1951).
8/ Payne, J. H., "Principles of Sugar Technology," Chap. 18, p. 766, P. Honig, Ed., Elsevier Pub. Co., Amsterdam (1953).
9/ McBurney, C. H., U. S. Patent 2,635,061 (1953).
10/ Kunin, R. and McGarvey, F. X., U. S. Patents 2,578,937 and 2,578,938 (1951).
11/ Juda, W. and McRae, W. A., J. Am. Chem. Soc. 72, 1044 (1950).

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The plastic columns used in the experiments are 3-1/8 inches in diameter and contain about 1/2 cubic foot of exchange resin in each column. Three of

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7/ Weller, H. A., U. S. Patents 2,388,194 and 2,388,195 (1945).
Naugle, J. J., Ind. Eng. Chem. 43, 649 (1951); Sugar 46 (1), p. 28 (1951).
8/ Payne, J. H., "Principles of Sugar Technology," Chap. 13, p. 765,
P. Honig, Ed., Elsevier Pub. Co., Amsterdam (1953).
9/ McBurney, C. H., U. S. Patent 2,635,061 (1953).
10/ Kunin, R. and McGarvey, F. X., U. S. Patents 2,578,937 and 2,578,938 (1951).
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the columns shown are used in series for each experiment, which is carried out with 30-40 gallons of clarified juice for a complete cycle. Commercial equipment for treating the juice of a factory grinding 2,500 tons of cane per day would be almost 1500 times the size of this experimental model. Such a large factory would require four beds of anion exchange resin, four beds of base-exchange resin, and at least three beds of the mixture of acid-exchange and decolorizing resins. Each resin bed would be 4 ft. deep in a 7-1/2 ft. diameter vessel, 8 ft. tall. Equipment must be acid and caustic resistant, and fitted for carrying out the steps of the cyclic process, preferably by automatic control of the flow of juice, water, and chemical solutions as required. This would provide for continuous treatment of clarified juice on its way to the factory evaporators.

Yield of Sugar

About 200 lbs. of ion-exchange sugar was produced experimentally during the 1954 grinding season with the small model equipment illustrated. Clarified juice from different lots of cane was obtained from the operation of a sugarcane processing pilot plant in cooperation with Louisiana State University at the experimental Audubon Sugar Factory in Baton Rouge, Louisiana. The ion-exchange process was operated intermittently so that juice from each cycle of ion-exchange purification could be evaporated to sirup separately. After the sirups were analyzed, they were combined for crystallization of first, second, and some third sugars. This was done in a laboratory scale vacuum pan 9 inches in diameter. The average results for twenty different lots of juice purified by ion-exchange showed that approximately 15 lbs. more sugar can be crystallized from the sirup for every ton of cane ground. The purity of clarified juices used averaged 82.7%. Average purity was raised to 90.0% by passing through the ion-exchange system. The proportion of sugar in the solids of the ion-exchange purified juice was increased by 8-9%, so that more sugar can be crystallized.

Quality of Sugar

The quality of the ion-exchange sugar is intermediate between that of standard granulated sugar and the colored turbinado sugar marketed for confectionery and other specialized uses. V lues for polarization, ash, color, and turbidity obtained by analysis of the grades of refined sugar and turbinado sugar being used in experimental candymaking, are compared with those for the ion-exchange sugar in the following table:

Some Analyses of Ion-Exchange Sugar
Compared with Granulated and Turbinado Sugars

	Granulated (Char Refined)	Ion-Exchange[a/]	Turbinado
Ash, parts per million	90	93	1000
Purity, percent[b/]	99.98	99.98	99.70
Color, transmittancy units[c/]	1.2	3.5	18
Turbidity, transmittancy units	5	32	64

a/ Blend of 1st, 2nd, and 3rd sugars.
b/ Estimated from ash content.
c/ Color and turbidity as -log t (transmittancy of 1 cm).

The ash and color approaches that of regular refined sugar, in which respect ion-exchange sugar is very much superior to the best grades of turbinado. Turbidity is comparatively high, and ion-exchange sugar would not be suitable for bottling. Its use is indicated particularly in candy-making, and in making ice cream, other confectionery items, and possibly in high sugar content jellies and preserves. It will have to be produced under conditions that will assure acceptable microbiological quality, with low counts of yeasts and molds.

Economics of Ion-Exchange Process

The equipment and exchange resins required for ion-exchange processes represent a considerable investment. Payne[8] estimated in 1951 that the cost would be $200 per ton of cane ground per day for equipment to carry out the process developed on a pilot plant scale in Hawaii. This is an investment of about half a million dollars for a factory grinding 2500 tons of cane per day, and is of the order of magnitude of the present cost of an ion-exchange plant using the new resins to carry out the present process. Amortization of this investment in a reasonable time must be taken into account in preliminary estimates of the cost of producing direct consumption sugar by ion-exchange.

Data obtained in a limited number of intermittent experiments on the small scale of operation used thus far are inadequate for the calculation of exact operating costs. They are indicative of the feasibility of the process and the soundness of larger scale pilot plant and development work. Continuous operation of a larger pilot plant to establish exact operating costs, yields, and optimum conditions will be more costly than the small scale experiments.

Approximate estimates, including capital investment and fixed costs, conservative operating costs, and allowance for the production of only half as much final molasses, indicate that ion-exchange purification can be employed profitably to produce a direct consumption sugar selling at about 1/2 cent per pound less than standard granulated sugar. The increased gross return to cover costs and a reasonable profit is derived both from the additional 15 pounds of sugar obtained per ton of cane, and from the increased value of all of the sugar which is of higher quality and suitable for direct consumption. Average recovery of sugar in Louisiana ranges from 150-165 lbs. per ton of cane. As raw sugar this brings a gross return of about $9.00 to $10.00 per ton of cane. Average production of about 7 gallons of molasses is worth an additional 60¢, or a total of $9.60-$10.60 per ton of cane ground. Ion-exchange purification can yield 160-180 pounds of direct consumption sugar which, at 7.5¢ to 7.75¢ per pound, would be worth $12 to $14 per ton of cane. Only about 3-1/2 gallons of molasses would be obtained, making the total gross return from this operation about $12.30 to $14.30 per ton of cane ground. The increase in gross return by ion-exchange purification would range from $2.70 to $3.70 per ton of cane.

Experimental Candy Production

First, second, and third sugars obtained in the experiments were blended to produce a uniform grade for use in preliminary candymaking trials.

Research on candymaking has been carried out at the Southern Regional Research Laboratory for the past 10 years in cooperation with the National Confectioners' Association. A complete experimental candy laboratory with facilities for making all types of candy in 5-10 lb. batches is operated by a chemist of the Laboratory staff and a candymaker whose services are provided by the Association.

Experimental batches of clear, hard candy, grained mints, grained marshmallow nougat, and starch and pectin jellies have been made with the blended ion-exchange sugar. In each case comparable batches of the candies were made of the same formulas in which regular granulated sugar was used. Flavors were used, but no colors were added in order to compare the results with the two different sugars in clear, or white candies. The ion-exchange sugar is a "strong" sugar for candymaking and no changes in formulation or procedure, nor any special precautions were required in making the different types of candy.

Color and texture qualities of all the candies were entirely acceptable. The turbidity in the ion-exchange sugar is dispersed in cooking so that the clarities of clear, hard candy and pectin jelly were equal to those of the comparable candies made with regular granulated sugar. Slight differences in color of the finished candies could be detected by careful comparison of the ion-exchange and granulated sugar pieces, but in no case was the candy made with ion-exchange sugar sufficiently different in color to affect its acceptability. No differences have been observed to date in graining, loss of moisture, or deterioration of texture of the candies in ordinary storage. Additional experiments are to be made in producing brilliant or light-colored candies of different popular types. The ion-exchange sugar is not being tried in chocolate bars or coatings, or highly colored candies such as fudge and caramel, for which turbinado sugar is satisfactory for most economical production.

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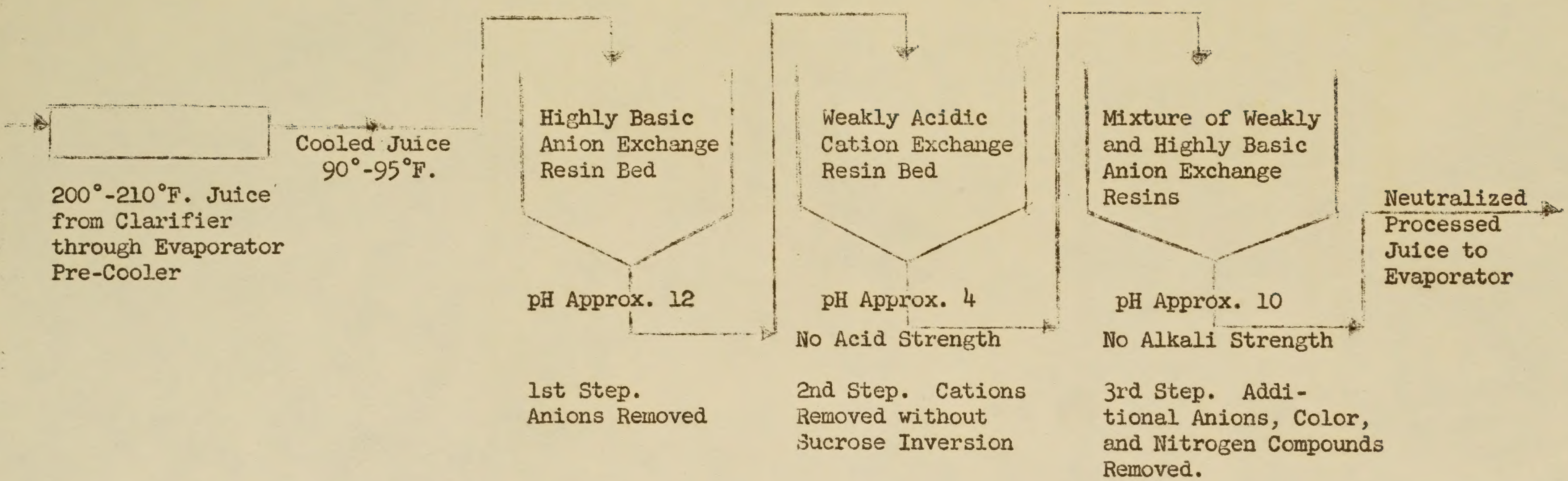
ION EXCHANGE TREATMENT OF CANE JUICE
(BY REVERSE CYCLE)



PROCESS ADVANTAGES:

1. Sucrose Inversion Loss of Older Demineralization Method Eliminated.
2. 8% More Sugar in the Bag per Ton of Cane.
3. Ash Content of Washed "A" and "B" Sugar Mixture in Refined Sugar Range; Ash Content of Washed "C" Sugar about 25% of a Washed Raw.
4. Over 80% of Juice Color and Turbidity Eliminated. Sugar Color Approximates That of Refined Sugars.
5. Only 1/2 Final Molasses Production of Raw Sugar Manufacture.